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NASA CR 121706

NBS REPORT

9797

FORTY-SECOND PROGRESS REPORT

to

National Aeronautics and Space Administration

on

Cryogenic Research and Development

Period Ending June 30, 1971



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**U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS**

Institute for Basic Standards
Boulder, Colorado 80302

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NBS PROJECT

27500-2750400
27503-2750430
27503-2750438
27505-2750451
27505-2750550
27506-2750460

June 30, 1971

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NATIONAL BUREAU OF STANDARDS

TABLE OF CONTENTS

	Page
1. Cryogenic Properties of Solids	1
1.1 Thermal Conductivity of Solids	1
1.1.1 General Comments	1
1.1.2 Program Status	1
1.2 Electrical Resistivity	2
1.2.1 General Comments	2
1.2.2 Program Status	2
2. Slush Hydrogen Heat Transfer and Hydrocarbon Suspension	8
2.0 General Comments	8
2.1 Slush Hydrogen Heat Transfer	8
2.2 Mixing Slush Hydrogen	8
2.3 Hydrocarbon Suspension in Slush Hydrogen . . .	9
2.3.1 Experimental Apparatus and Procedure	9
2.3.2 Test of Hydrocarbon Suspensions	10
3. Slush Hydrogen Instrumentation	12
3.0 General Comments	12
3.1 Carbon Film Sensors	12
4. Consultation and Advisory Services	16
4.0 General Comments	16
4.1 NERVA Program	16

Task Completed		
For Latest Report or Publication on This Task See	Provisional (P) or Final (F)	
	item #	
	page	
	Quart. Report #	
Est. Completion Date		* 11/1/71 1/1/72 1/1/72 *
Report in Press		x
Report Composition		x
Data Analysis		x x x
Data Taking		x x x
Apparatus Construction		x
Apparatus Designing		x
Preliminary Planning		x
Not Started		
Active Coord. with Lewis, MSFC, LASL		
<div> <div>PHASE OF TASK →</div> <div>TASK ↓</div> </div>		<div> <div>Cryogenic Properties of Solids</div> <div>Thermal Conductivity</div> <div>Electrical Resistivity</div> <div>Slush Hydrogen</div> <div>Heat Transfer</div> <div>Hydrocarbon Suspension</div> <div>Slush Hydrogen Instrumentation</div> <div>Consultation & Advisory Services</div> <div>NERVA Program</div> </div>
Item Number		<div> <div>1.</div> <div>1.1</div> <div>1.2</div> <div>2.</div> <div>2.1</div> <div>2.2</div> <div>3.</div> <div>4.</div> <div>4.1</div> </div>

* Continuing effort

1. Cryogenic Properties of Solids

1.1 Thermal Conductivity of Solids

1.1.1 General Comments

The objectives of this program are (1) to measure the thermal conductivities of several standard reference materials, (2) to study Lorenz ratio of several classes of materials, and (3) to study the conduction mechanisms in alloys in the temperature range from 4 to 300 K.

Personnel contributing during the reporting period were J. G. Hust and L. L. Sparks.

1.1.2 Program Status

During the current period the Lorenz ratio compilation has been nearly completed. The literature search is complete, references have been received, and data are now being punched on cards for computer analysis. This analysis will be completed during the next reporting period.

Measurements have been completed on both lots of AGARD stainless steel. We have found that hot swaging and reannealing does not alter the thermal conductivity of this steel. Therefore we are proceeding with specimen preparation for distribution by OSRM. The specimen preparation is being done by USBM, Albany, Oregon. An additional 200 pounds of this steel have been ordered from the German supplier. A report will be completed during the next period describing the results of characterization and measurements.

Also, during the next period we will continue to analyze the heat conduction mechanisms of the alloys measured in the past.

1.2 Electrical Resistivity

1.2.1 General Comments

The objectives of this project are to measure the low temperature electrical resistivity of selected alloys needed for cryogenic design and to study their material variability, and to perform precision measurements of resistivity for several high purity metals to improve understanding of the basic electron scattering mechanisms and to improve the reliability of standard reference data.

The material variability program has been completed and is reported. A. F. Clark contributed during this reporting period.

1.2.2 Program Status

Nearly 50 measurements were performed for the material variability program and are reported along with the previous measurements in Tables 1.2.1 - 1.2.5. Analysis of the data has been completed and an article has been written for submission to Cryogenics. A preprint of this article will be appended to the next progress report.

During the next reporting period the measurement of the precision electrical resistivity of aluminum will be initiated.

Table 1.2.1 Low Temperature Electrical Resistivity - Al 2024

VARIABLE		CONDITION	RESISTIVITY $\mu\Omega m$				
Mfg.	Heat	Within Heat	273 K	192.4 K	75.75 K	19.65 K	4.0 K
AS RECEIVED CONDITIONS							
		A23a					
		A23b					
		A23c					
		A23d					
(A23)		(Av of A23a-d)					
A21		Unknown (Prob. 0)					
A22		Unknown (Prob. 0)					
A24		Unknown (Prob. 0)					
(A2)		(Av of A21-4)					
A1		T4					
9		T86					
A3		Unknown (Prob. T4)					
SEE BELOW							
HEAT TREATED CONDITIONS							
		A23a					
		A23c					
(A23)		(Av A23a and c)					
A21		"0"					
(A2)		(Av A21 and 23)					
9		"0"					
A1		"0"					
(Av-0)		(Av of A2, 9, and A1)					
9		T4 (After Ann.)					
A1		T4					
A21		T4					
A3		Unknown (Prob. T4)					
(Av-T4)		(Av of 9, A1, A21, A3)					
9		T6					
A1		T6					
A21		T6					
(Av-T6)		(Av of 9, A1, A21-T6)					

Table 1.2.2 Low Temperature Electrical Resistivity - Al 5000 series

VARIABLE		CONDITION	RESISTIVITY $\mu\Omega\text{m}$				
Mfg.	Heat		273 K	192.4 K	75.75 K	19.65 K	4.0 K
	Within Heat						
	A53a	Unknown; 5056	.05644	.04697	.03362	.03080	.03076
	A53b	Unknown; 5056	.05649	.04697	.03360	.03077	.03074
	A53c	Unknown; 5056	.05641	.04699	.03357	.03074	.03073
	A53d	Unknown; 5056	.05617	.04679	.03342	.03060	.03057
(A53)		(Av of 53a-d)	(.05638)	(.04693)	(.03355)	(.03073)	(.03070)
A51		Unknown; 5056	.05662	.04722	.03370	.03087	.03085
A52		Unknown; 5056	.05699	.04729	.03455	.03168	.03152
A54		Unknown; 5056	.05592	.04642	.03285	.03002	.02999
(A5)		(Av of A51-4)	(.05648)	(.04697)	(.03366)	(.03083)	(.03077)
A4		Unknown; 5083	.05591	.04640	.03288	.03007	.03003
7		H 113 across; 5083	.05711	.04741	.03362	.03069	.03065
A6		Unknown; 5456	.05918	.04946	.03556	.03264	.03261
(Av)		(Av of A4-6, and 7)	(.05717)	(.04756)	(.03393)	(.03106)	(.03102)
7		Annealed; 5083	.05658	.04694	.03319	.03032	.03030
8		H 113 with; 5083	.05683	.04715	.03344	.03054	.03052

Table 1.2.3 Low Temperature Electrical Resistivity - Inconel 718

VARIABLE		CONDITION	RESISTIVITY $\mu\Omega m$					
Mfg.	Heat	Within Heat	273 K	192.4 K	75.75 K	19.65 K	4.0 K	
ANNEALED								
		I1a	Hot Rolled Annealed	1.2585	1.2382	1.2085	1.2035	1.2098
		I1b	Hot Rolled Annealed	1.2579	1.2376	1.2082	1.2032	1.2095
		I1c	Hot Rolled Annealed	1.2548	1.2344	1.2048	1.1996	1.2058
(I1)			(Av of I1a, b, c)	(1.2571)	(1.2367)	(1.2072)	(1.2021)	(1.2084)
I2			Hot Rolled Hot Rolled	1.2623	1.2421	1.2157	1.2115	1.2178
I3			Hot Rolled Annealed	1.2429	1.2257	1.2020	1.1993	1.2056
I4			Hot Rolled Annealed	1.2438	1.2263	1.2015	1.1977	1.2038
22			Ann. (After Aging)	1.2553	1.2376	1.2128	1.2104	1.2173
24			Ann. (After Aging)	1.2254	1.2137	1.1854	1.1825	1.1884
(Av)			(Av of I1-24)	(1.2478)	(1.2304)	(1.2041)	(1.2006)	(1.2069)
AGED								
		I1a	Aged	1.0929	1.0644	1.0193	1.0100	1.0137
		I1b	Aged	1.0891	1.0606	1.0156	1.0064	1.0101
		I1c	Aged	1.0987	1.0703	1.0263	1.0170	1.0208
(I1)			(Av of I1a, b, c)	(1.0936)	(1.0651)	(1.0204)	(1.0111)	(1.0149)
I2			Aged	1.1582	1.1324	1.0922	1.0839	1.0881
I3			Aged	1.1677	1.1423	1.1041	1.0965	1.1008
I4			Aged	1.1289	1.1014	1.0592	1.0502	1.0539
22			Aged by Mfg.	1.1449	1.1205	1.0839	1.0775	1.0815
24			Aged by Mfg.	1.0655	1.0357	0.9925	0.9839	0.9882
(Av)			(Av of I1-24)	(1.1265)	(1.0996)	(1.0587)	(1.0505)	(1.0547)

Table 1.2.4 Low Temperature Electrical Resistivity - A286 Stainless Steel

VARIABLE		CONDITION	RESISTIVITY $\mu\Omega m$			
Mfg.	Heat		273 K	192.4 K	75.75 K	19.65 K
ANNEALED						4.0 K
(S4)	Within Heat	S4a	.9294	.8753	.7927	.7695
		S4b	.9298	.8766	.7934	.7704
		S4c	.9298	.8765	.7938	.7709
		S4d	.9298	.8759	.7924	.7693
		(Av of S4a -d)	(.9297)	(.8761)	(.7931)	(.7700)
	S7a	Solution Annealed	.9365	.8839	.7989	.7745
	S7b	Solution Annealed	.9371	.8849	.8008	.7758
	S7c	Solution Annealed	.9466	.8945	.8132	.7907
	S7d	Solution Annealed	.9383	.8854	.8015	.7774
(S7)		(Av of S7a -d)	(.9396)	(.8872)	(.8036)	(.7796)
S1		Annealed (After Aging)	.9251	.8707	.7835	.7604
S2		Solu. Ann. C. D. 15%	.9357	.8823	.7950	.7745
S3		Annealed (After Aging)	.9474	.8945	.8097	.7868
(Av Ann.)		(Av of S1 -4, and S7)	(.9355)	(.8822)	(.7970)	(.7743)
	S4b	Re-Annealed	.9281	.8740	.7883	.7661
	S4c	Re-Annealed	.9267	.8728	.7873	.7650
	S7a	Re-Annealed	.9356	.8823	.7954	.7714
	S7b	Re-Annealed	.9377	.8841	.7973	.7727
AGED						
S1		Aged (As Recd)	.8846	.8193	.7169	.6912
S3		Aged (As Recd)	.9234	.8731	.7937	.7711
S7a		Aged (After Reann.)	.9032	.8430	.7436	.7154
(Av Aged)		(Av of S1, S3, S7a)	(.9037)	(.8451)	(.7514)	(.7259)
S3		Aged (After Ann.)	.9094	.8502	.7536	.7268

Table 1.2.5 Low Temperature Electrical Resistivity - AISI 316 Stainless Steel

VARIABLE		CONDITION	RESISTIVITY $\mu\Omega m$					
Mfg.	Heat		273 K	192.4 K	75.75 K	19.65 K	4.0 K	
		Within Heat						
	S5a	Hot Rolled-Redrawn	0.7679	0.7005	0.5890	0.5573	0.5590	
	S5b	Hot Rolled-Redrawn	0.7664	0.6988	0.5869	0.5561	0.5569	
	S5c	Hot Rolled-Redrawn	0.7663	0.6992	0.5866	0.5560	0.5568	
	S5d	Hot Rolled-Redrawn	0.7661	0.6993	0.5865	0.5559	0.5567	
(S5)		(Av of S5a, b, c, d)	(0.7667)	(0.6995)	(0.5873)	(0.5563)	(0.5574)	
	S6a	Cold Drawn	0.7574	0.6897	0.5763	0.5465	0.5470	
	S6b	Cold Drawn	0.7634	0.6950	0.5832	0.5514	0.5518	
	S6c	Cold Drawn	0.7539	0.6861	0.5719	0.5410	0.5416	
	S6d	Cold Drawn	0.7564	0.6880	0.5749	0.5439	0.5444	
	51	Cold Drawn and Ann.	0.7644	0.6963	0.5856	0.5532	0.5534	
	85	Cold Drawn and Ann.	0.7654	0.6967	0.5852	0.5528	0.5531	
(S6+)		(Av of S6a -d, 51, 85)	(0.7602)	(0.6920)	(0.5795)	(0.5481)	(0.5486)	
86		Cold Drawn and Ann.	0.7757	0.7066	0.5950	0.5626	0.5628	
87		Annealed	0.7498	0.6794	0.5663	0.5383	0.5392	
88		Cold Drawn and Ann.	0.7184	0.6426	0.5232	0.5015	0.5029	
(Av)		(Av of S5, S6+, 86-8)	(0.7542)	(0.6840)	(0.5703)	(0.5414)	(0.5422)	

2. Slush Hydrogen Heat Transfer and Hydrocarbon Suspension

2.0 General Comments

Activity on the program during this reporting period has been concerned with completion of the slush mixing study, start up of the experimental apparatus to prepare a hydrocarbon suspension in slush hydrogen, and initiation of the design of the heat transfer experimental apparatus. Personnel contributing to the program were C. Sindt, P. M. McConnell, R. O. Voth, and J. Hord.

2.1 Slush Hydrogen Heat Transfer

The activity on this task has included selection of the basic design for the experimental heat transfer unit. The design selected is similar to that reported by K. J. Coeling and H. Merte, Jr. (1968). This design was selected because it has been used successfully for liquid hydrogen and will therefore provide some correlation data between systems as well as from slush and triple-point liquid to normal boiling liquid. The design heat flux values are estimated to range from the visible boiling threshold to the lowest values measurable on the heater circuit.

2.2 Mixing Slush Hydrogen

The study of mixing requirements and design for slush hydrogen systems has been completed. The study has resulted in selection of the most promising mixer configurations for one-g mixing and for low-g mixing. The completed study will be reported separately during the next quarter and will be titled "Slush Hydrogen Mixing: Preliminary Study".

2.3 Hydrocarbon Suspension in Slush Hydrogen

The apparatus for preparation and study of hydrocarbon suspension in slush hydrogen has been completed and two tests and a checkout run have been made using hydrogen.

2.3.1 Experimental Apparatus and Procedure

The experimental dewar is thermally protected by a liquid hydrogen bath dewar which can be vacuum pumped to triple-point pressure. Triple-point pressure is maintained in the bath dewar with a barostat that controls the pressure within 270 N/m^2 (2 mm Hg). The bath dewar is thermally protected with an open mouth, liquid nitrogen filled dewar. For visual and photographic observation, all of the dewars used are clear glass during the initial experiments. If a stable hydrocarbon suspension is obtained, the bath dewar will be replaced with a strip-silvered dewar to assure long observation periods of the suspension with a minimum of melting of the solid hydrogen in the slush.

The hydrocarbon content of the suspension is determined with a hydrogen flame hydrocarbon analyzer. The suspension sample is taken with a micro-sampler of approximately 1 cm^3 capacity, which can sample at any level in the experimental dewar. The sample which is contained in the fluid sampler is then raised above the liquid hydrogen level and boiled with electrical heaters. The resulting gas is captured in two evacuated chambers. The first chamber is filled to approximately one atmosphere pressure when the hydrogen has gasified. This chamber is then closed and the second evacuated chamber is opened. The second chamber is filled as the remaining hydrogen and the hydrocarbon are heated well above the boiling point of the hydrocarbon. Since the second chamber is over 300 times larger

than the sample volume, 99.7% of the gas mixture remaining after filling the first volume is contained in the second volume. This serves to extract essentially all of the gas sample from the sampler. The two volumes are then mixed with each other resulting in a gas sample typical of the fluid sample trapped in the micro-sampler.

2.3.2 Test of Hydrocarbon Suspensions

Three hydrogen tests have been made in the apparatus. The first test was conducted to check out operation and safety features. The second hydrogen test was conducted with ethane injection into triple-point liquid hydrogen. The injection gas was three volume percent ethane in hydrogen. The injection time was 30 seconds with the pressure at 430 kN/m² (50 psig) and the injection was through a 0.33 mm diameter hole. Solid ethane particles which appeared to be as large as 5 mm formed during injection and the particles were of the texture of snow flakes. Hydrogen slush was made after injection. During slush preparation, the mixture was stirred vigorously. After the solids had settled the mixture appeared to contain solid ethane throughout the slush hydrogen. The ethane particles are discernible from solid hydrogen because they are white and the solid hydrogen has the appearance of a wetted translucent particle.

Attempts were made to sample the clear liquid over the settled solids and to sample in the settled solids region. The samples in the settled portion were approximately 700 ppm by weight of ethane; however, the sampler was not functioning properly so this value is suspect. The clear liquid samples contain no detectable ethane.

The mixture of slush and hydrocarbon was stirred on several occasions and the solids always appeared to settle with the ethane well dispersed in the slush. No solid ethane particles were visible in the liquid after settling had occurred. Because of the sampler problem the experiment was terminated without adding more ethane. After the solid hydrogen in the slush had melted the solid ethane appeared to occupy about 1/20 of the liquid volume.

The third experiment was terminated before data were available because of a heater failure.

Since the third experiment, the heaters and the micro-sampler have been modified to increase reliability and to improve the accuracy of the sample.

Future experiments will include more testing with 10% cyclopropane in hydrogen as the injection gases. Experiments where the hydrocarbon is to be added after slush is made will use pure methane as the injection gas initially. The injection gas must be of high hydrocarbon content as the large volume of warm hydrogen gas associated with diluted gas injection will melt a significant portion of the solid hydrogen.

3. Slush Hydrogen Instrumentation

3.0 General Comments

Contributing to the program during this reporting period was R. S. Collier.

3.1 Carbon Film Sensors

Analysis of the thermal oscillations in slush was begun using the heat and mass transport equations. If the viscosity of the fluid and the slush is neglected there are six relevant quantities at each point in the fluid — namely, the three fluid velocity components, \vec{v} ; the density, ρ ; the pressure, P ; and the temperature, T . In order to solve for any of these quantities for any point in the fluid as a function of time (the Eulerian point of view), it is necessary to establish six independent relations between the quantities. The conservation of momentum gives three independent relations, the conservation of mass and energy gives two more, and the last relation is obtained from the equation of state which related P , ρ , and T in a state of thermodynamic equilibrium. Since the thermal oscillations are low frequency (approximately 8 Hz) and the thermal amplitudes are small (approximately 0.1 K), we shall assume that the equation of state holds locally even though the system is not in thermal equilibrium.

$$\rho \frac{D\mathbf{v}}{Dt} = -\nabla P \quad (1)$$

where D/Dt is the convective derivative $\partial/\partial t + \mathbf{v} \cdot \nabla$. The conservation of mass is expressed as

$$\frac{\partial}{\partial t} \rho + \nabla \cdot \rho \mathbf{v} = 0. \quad (2)$$

To a first approximation, the slush can be treated as an incompressible fluid. Under this assumption and considering spherical symmetry of

the pressure and velocity waves about the film sensor, equations (1) and (2) can be integrated in the space variable to give

$$R \frac{d^2 R}{dt^2} + \frac{3}{2} \left(\frac{dR}{dt} \right)^2 = \frac{1}{\rho} (P_R - P_\infty) \quad (3)$$

where R is the radial position of a fluid particle, P_R is the fluid pressure at R , and P_∞ is the ullage pressure. Using some simple approximations relating P_R to R we have found the conditions for which the computer solutions to equation (3) are oscillatory. The next step is to relate these oscillations to the thermal oscillations using the energy equation, the Equation of State, and the boundary conditions giving the heat input. This work is continuing and the full details will be given in a future report.

Work was begun with Metro Physics, Inc., Santa Barbara, California, to transfer our carbon film technology and to obtain a source of carbon films which will be used as slush sensors, thermometers, and fluid phase detectors.

Six deposition runs were made using eight film substrates per run. The first problem was to establish a deposition rate which would be held constant on succeeding runs. The deposition rate had to be slow enough to make stable films, yet high enough to make a 500Å - 1000Å film in a reasonable length of time. (This rate had been previously determined by NBS research to be about 0.5 - 2.0 Å/min.) The rate of thickness was determined by a shielded quartz crystal monitor. The crystal monitor was placed 8 inches from the source and the substrates 24 inches from the source. Using the $1/R^2$ law and figuring the rotation of the substrate, there is a factor of 9π between the rate of the crystal

monitor and the rate at the substrate. We therefore initially kept the rate at the crystal monitor about 60 Å/min for a flat non-rotating substrate. At this rate we were able to deposit 600 Å films in about five hours, which gave sensor resistance of 300 - 1500 Ω at room temperature (the range being dependent on the substrate temperature). At this rate, the rotating substrates had films which were of excellent optical quality, were stable, and reproducible to within 0.1 percent on thermal cycling (more sensitive measurements will be forthcoming). However, the flat substrates at 6 Å/min were all unstable.

The nominal rate of 60 Å/min at the quartz crystal monitor was controlled by the emission current of the electron beam gun which had a tendency to drift and required a correction at intervals of about 10 minutes. The rate then was usually kept between 50 - 70 Å/min, although it would occasionally drop as low as 40 Å/min or get as high as 80 Å/min. An automatic emission current controller would be very helpful here, but may require very sophisticated circuitry as there are time lags of 4 or 5 minutes to changes in emission current.

The next problem was to control the substrate temperature and measure the effect of substrate temperature on the total resistance and sensitivity (as measured by the resistance ratio between room temperature and liquid nitrogen boiling point). The first attempt at reproducibility gave very good results (to within 10 percent in sensitivity and resistance) although the sensitivity seemed to be a little too high. The next two runs were unsuccessful in establishing a definite trend of sensitivity vs. substrate temperature and we began to notice a correlation with respect to substrate position. The final two runs, after eliminating the effect of all other parameters, showed that the relevant parameter seemed to be the warm up time. A

hypothetical reconstruction of events goes as follows: The substrates are mounted axially on beveled rollers about 2 inches in diameter and 2 inches long. Eight rollers fit in a thrust bearing and rotate on a 12-inch diameter circle. The bottom bearing is heavy aluminum (called the track) and the top bearing is heavy aluminum (called the wheel). The rollers roll between the wheel and the track. The wheel is warmed from above by quartz lamps and the track is warmed from below by quartz lamps and the carbon source. However, the rollers are warmed primarily by conduction at the points of contact between the track and wheel. It was necessary to give the rollers a heavy coat of moly-disulfide to prevent sticking, probably making a poor thermal contact. Thus, it may take longer than the 3 or 4 hours which were allowed to come to thermal equilibrium. The temperature of the wheel and track was monitored, but the roller temperatures are difficult to determine. This hypothesis seems to explain everything that we observed, including the location correlation and the first attempt at reproducibility which had very nearly the same warm up time.

It seemed reasonable to stop the calibration runs temporarily and do some separate experiments to instrument one or two of the rollers in a static test to determine the warm up time. Metro Physics will do this in the next week or two and will also build a controller which will allow long warm up times, say, over the weekend. If this experiment is successful, it would be desirable to do three or four more calibration runs where more information is known about the actual substrate temperature.

4. Consultation and Advisory Services

4.0 General Comments

Contributing personnel were A. F. Schmidt and H. M. Roder.

4.1 NERVA Program

During the reporting period, various organizations were provided information on hydrogen properties data: (1) NBS Reports 9288 and 9711 were furnished S. Wilkes (Martin-Denver); (2) NBS Report 9288 and a hydrogen properties TAB deck were sent to R. F. Hausman (Lockheed-Sunnyvale); (3) NBS Report 9711 was requested by Mrs. M. Lura (Sandia Corporation); (4) a high temperature hydrogen generating function deck was sent to R. A. Silver (Edwards Air Force Base, Calif.); (5) para and equilibrium hydrogen properties diagrams were discussed with C. L. Carwile (SNSO-C); (6) references for high temperature hydrogen properties were given to S. Manatt (AiResearch Corp.). Also, helium properties charts and tables were provided SNSO-C, and discussions on several topics were held with SNSO-W.

The NERVA Engine Design Status Review Meeting was attended May 11-13 at Aerojet Nuclear Systems Company, Sacramento, California. On June 13-14, meetings were held in Boulder with Dr. Landon Nichols concerning both present and proposed work under NASA (SNSO-C) Contract R-45.

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